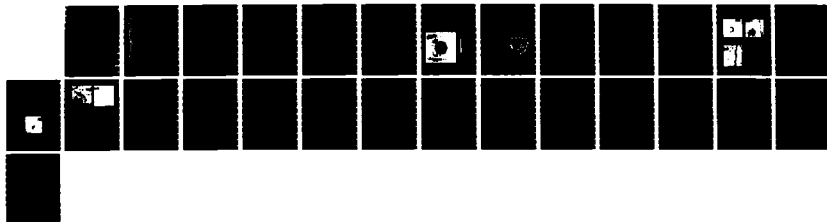
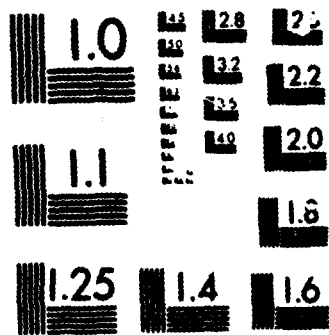


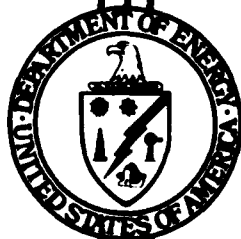
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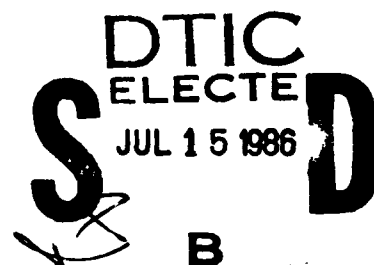
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MECHANICAL PROPERTY EVALUATION AT ELEVATED TEMPERATURES OF SINTERED BETA SILICON CARBIDE

MICHAEL J. SLAVIN and GEORGE D. QUINN

March 1986



Prepared under
Interagency Agreement DE-A105-84OR-21411

by
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ABSTRACT

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A program was carried out to evaluate the mechanical properties at room and elevated temperatures of the General Electric sintered beta silicon carbide. The testing included room temperature flexural strength, flexural stress rupture at 1200°C, and stepped temperature stress-rupture (STSR) experiments. Fractographic examination identified the flaw populations that limited strength. The properties measured on this material are typical of sintered silicon carbide.

Keywords: Microhardness

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INTRODUCTION

↙ Silicon-based ceramics have considerable potential for structural applications in heat engines and other energy conversion devices. This study is part of an on-going Department of Energy project to screen new materials for their mechanical properties for heat engine applications.

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This report presents findings on a commercially available sintered beta silicon carbide manufactured by General Electric Company. In ten years of development, this material has evolved from a hot-pressed laboratory grade material to a commercial-sintered product.¹⁻⁷ It is fabricated from a submicron (beta) silicon carbide powder with small additions (≈ 0.5 wt% each) of boron and carbon which is sintered in a reducing atmosphere at approximately 2100°C. The boron promotes diffusion, while the carbon acts to reduce possible oxide phases (SiO_2) and retard grain growth. The sintered product often contains some alpha phase silicon carbide as a result of phase transformation during sintering. This latter process is dependent upon impurities and sintering conditions and can be very sensitive to conditions of powder preparation, processing, and densification.³ The presence of the alpha phase is not desired because it can manifest itself as large tabular grains that can limit strength.

Earlier studies have suggested that the laboratory hot-pressed and sintered beta silicon carbide materials have excellent high temperature properties up to 1500°C, including creep and slow crack growth resistance⁸⁻¹⁰ and strength retention.^{1,4,9,10} These favorable properties, coupled with the complex shape capabilities of sintering, suggest that General Electric's beta silicon carbide is a promising heat engine grade ceramic. The purpose of this study was to verify these properties in the commercial form of the material.

MATERIAL

The sintered beta silicon carbide was supplied by General Electric Company's Manufacturing Division, Houston, Texas, in the form of eight 5.08 x 5.08 x 1.27-cm (2 x 2 x 0.5-in.) tiles in March, 1984. Four of these tiles were machined into flexure specimens according to MIL-STD-1942(MR),¹¹ specimen configuration "B." Specimen size was 3.0 x 4.0 x 44.5 mm with a 45° chamfer along the four long edges. Chamfer depths

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11. U.S. Army MIL-STD-1942(MR). *Flexural Strength of High Performance Ceramics at Ambient Temperature*.

were inadvertently machined to approximately 0.28 mm which is greater than the 0.15 ± 0.05 mm specified in the standard. Although this causes only a 1.25 percent decrease in the cross-sectional area, the strengths reported in this study (based on elastic theory with a rectangular cross section) are 3.4 percent lower.

The average bulk density of the tiles was 3.12 g/cm^3 , with a standard deviation of 0.03 and is in agreement with the 3.12 g/cm^3 reported. The elastic modulus and Poisson's ratio as measured via the sonic method were 395 GPa and 0.17, respectively.

A powerful characterization tool is the ultrasonic longitudinal wave propagation time C-scan. The time C-scan shows spatial variation of wave propagation times through the tile thickness with a resolution of ± 1 nanosecond. If a tile has constant thickness, the C-scan can be interpreted as a longitudinal velocity C-scan. Figure 1 is a C-scan of a $2 \times 2 \times 1/2$ -inch tile of beta silicon carbide and, allowing for wave distortion near the edges, had a variation in wave propagation time of approximately 5.5 percent. In the Appendix is an analysis of the C-scan in which the relationship between the tile's thickness, elastic modulus, Poisson's ratio, and density as they pertain to the longitudinal wave velocity is evaluated.

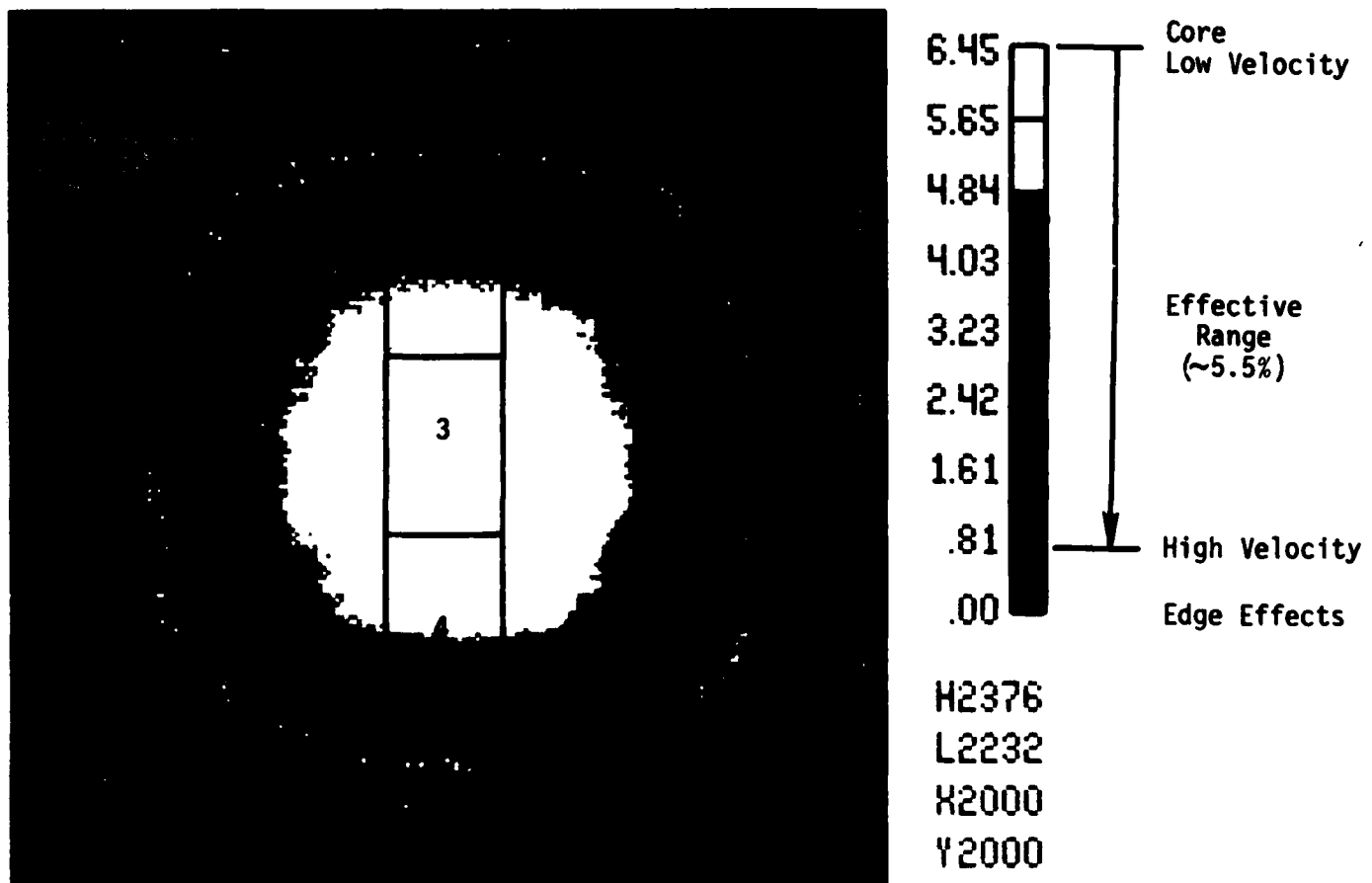


Figure 1. Time-of-flight C-scan for General Electric sintered beta silicon carbide using a 10 MHz undamped transducer.

The increase in the wave propagation time, from the edge to the center of the tile, is due to (in order of importance): an increase in thickness, a decrease in elastic modulus, and a decrease in Poisson's ratio. The density, which decreases toward the tile's center, decreases the wave propagation time.

A quantitative analysis of the metallic impurities is given in Table 1. A polished section showing the material porosity ($\approx 3\%$) is shown in Figure 2. Figure 3 shows the same section etched with boiling Murikami's reagent to delineate the grain boundaries.⁶ Tabular grains commonly associated with the alpha phase of silicon carbide¹⁻⁵ are readily apparent. X-ray diffraction of the silicon carbide showed that the majority of the material is beta silicon carbide with a small percentage of the alpha phase and a small amount of graphite. In a parallel study being carried out at MTL* for nonheat engine applications, a 15.24 x 7.62 x 1.27-cm (6 x 3 x 1/2-in.) tile had an identical X-ray diffraction pattern and a very similar microstructure (larger alpha grains).

Table 1. IMPURITY OR ADDITIVE CONTENT OF THE GENERAL ELECTRIC SINTERED BETA SILICON CARBIDE. WEIGHT PERCENTS DETERMINED BY EMISSION SPECTROSCOPY.

Elements												
Al	B	Ca	Co	Cr	Cu	Fe	Mg	Mn	Ni	Ti	Zr	V
0.008	0.5	0.017	*	0.005	0.005	0.003	0.002	~ 0.001	0.002	0.004	0.16	*

*Not detected

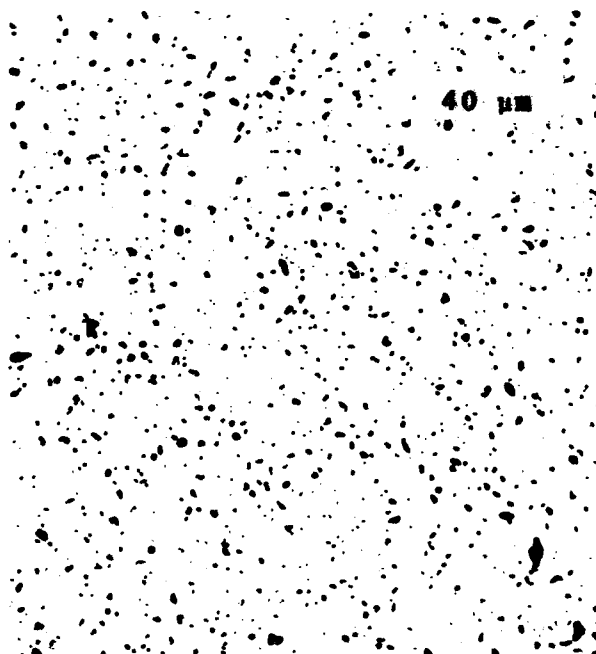


Figure 2. Polished section of a specimen of beta silicon carbide showing the distribution of porosity.

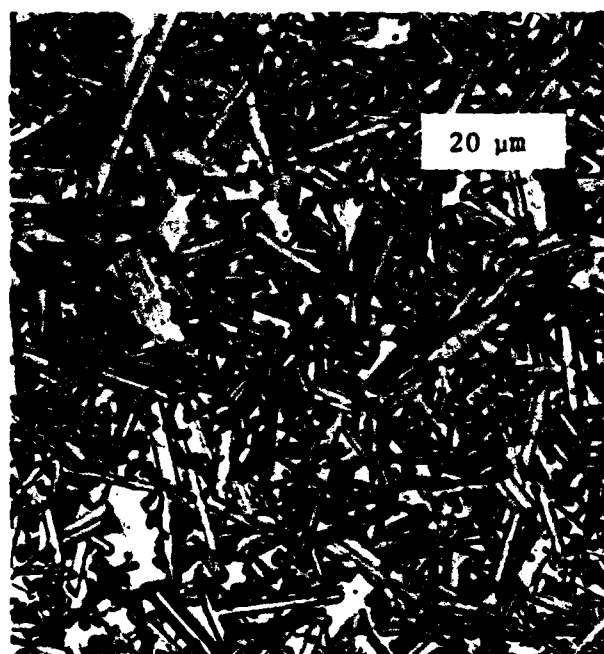


Figure 3. Etched section of a specimen showing grain size distribution. Note the long tabular alpha grains.

*Tracy, C. A., unpublished research, U.S. Army Materials Technology Laboratory (MTL), 1984.

PROCEDURE

Microhardness testing was performed on a mounted and polished specimen with a load of 2000 gf (19.6N). Using both Vickers and Knoop indentors, 10 indentations each were made with a standard microhardness indenter.* Fracture toughness was estimated from crack lengths emanating from Vickers indentations at loads of 2000 gf (19.6N), 3000 gf (24.9N), and 3500 gf (34.3N), using a standard microhardness indenter.†

Room temperature flexural testing was used to establish a reference strength and to characterize the strength-limiting flaws. A universal testing machine† was used with four-point fixtures in accordance with MIL-STD-1942(MR).¹¹ The spans were 4.00 cm and 2.00 cm and the crosshead speed was 5.0×10^{-2} cm/min. The relative humidity was 35 percent at 23°C.

The 1200°C flexural stress-rupture experiments were carried out in air in several test furnaces. These furnaces were constructed with firebrick and employed silicon carbide heating elements as the heat source. Four-point bend fixtures, machined from hot-pressed silicon carbide, having fixed bearing spans of 3.81 cm and 1.90 cm were used. A simple deadweight lever system applied the load into the furnace. The temperature inside the furnace was allowed to stabilize for five minutes prior to loading. Additional details of this procedure are reported in References 12 and 13.

A stepped temperature stress-rupture (STSR)¹⁴ program was used to quickly screen the material while stressed to a range of temperatures that it may be subjected to in an engine environment. This program assesses where creep or static fatigue may be a problem. The furnace arrangement and loading procedure are identical to the stress-rupture tests. The program starts at 1000°C when the load is applied. After 24 hours, if the specimen survives, the temperature is raised (within approximately 1/2 hour) to 1100°C. This cycle continues for 1200, 1300, and 1400°C, but at 1400°C the specimen cycle time is 72 hours. In the event of a failure, the furnace is shutdown. An arrow labeled with the applied stress denotes the time of failure on the STSR plot.

Specimens that survived the stress-rupture or STSR tests were unloaded prior to cool-down. This was done to minimize frictional forces at the knife edges due to unequal specimen-fixture contractions. The survivors were then tested at room temperature to determine the retained strength. They were mounted in the room temperature fixtures such that the most highly stressed zone in the high temperature test was again loaded in a tensile manner. Note that the room temperature fixtures have slightly larger inner and outer spans than the high temperature fixtures.

*Miniload II, Leitz, W. Germany

†Tukon Tester FB, Wilson Mechanical Instrument Co. Inc., NY

††Instron Corp., Model TT-DL, Canton, MA

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RESULTS

The Vickers indentations produced an average hardness of 23.9 GPa (2430 g/mm²) with a standard deviation of 0.4 GPa (40 g/mm²) and the Knoop indentations produced an average hardness of 20.9 GPa (2129 g/mm²) with a standard deviation of 0.3 GPa (30 g/mm²). This is somewhat lower than the HV1000 gf of 28.0 GPa (2850 g/mm²) reported in Reference 7, but a little higher than the average HK1000 gf of 20.1 GPa (2050 g/mm²) with a standard deviation of 0.8 GPa (80 g/mm²) (60 indentations) in the unpublished research by Tracy of MTL.

The fracture toughness values were measured from Vickers indentations and evaluated using the Marshall-Evans formulations.¹⁵ A minimum of five "good" indentation patterns were obtained for each of the three loads. Some problems with spall and cracks not radiating from the corners were observed. A "good" indentation pattern consisted of at least three of the four cracks starting from the corners and extending straight from the indent and no spall. Table 2 indicates the K_{IC} values which averaged 2.8 MPa m^{0.5}. This is somewhat lower than other reported values: 3.0 MPa m^{0.5} (double torsion),¹⁶ 3.3 MPa m^{0.5} (single edge notch beam)* (unpublished research by Tracy of MTL), and 3.1 MPa m^{0.5} (controlled flaw in flexure).¹⁷

Table 2. FRACTURE TOUGHNESS (K_{IC}) FOR VARYING INDENT LOADS.*

Load (gf)	Number of Indents (n)	$K_{IC0.5}$ (MPa m ^{0.5})
2000	5	2.82
3000	6	2.81
3500	6	2.78

*Average K_{IC} = 2.8 MPa m^{0.5}, with a standard deviation of 0.2.

Thirty room temperature four-point fast fracture specimens were tested and had an average flexure stress of 346 MPa (50.2 ksi) with a standard deviation of 34 MPa (5 ksi). This average is appreciably less than values for the laboratory prepared three-point flexure specimen in Reference 6, is somewhat less than the 440 MPa (63.8 ksi) four-point results reported in Reference 9 and 10, is also less than the three-point value of 533 MPa (77.3 ksi) in Reference 7, and is quite consistent with the four-point value of 389 MPa (56.4 ksi) with a standard deviation of 52 MPa (8 ksi) in the unpublished research by Tracy of MTL.

A Weibull two-parameter modulus was determined by a least-squares fitted line through the strength data when plotted on a typical Weibull format (see Figure 4). The ranking percentage is $p=i/(N+1)$, where i is the i th data point and N is the total number of specimens. The Weibull modulus, which is the slope of the line, is 11.0 and has a correlation coefficient of 96.5 percent. Figure 5 is a Weibull plot for the strength data from the parallel study underway at MTL by Tracy for nonheat engine applications. The Weibull modulus is 8.1 with a correlation coefficient of 98.8 percent. These moduli are consistent with the reported value of 8 to 15 in Reference 7.

*Single edge notched beam, 1/2 - 4, 4.0 cm outer span, cross rate of 0.5 mm/min, specimen size 5.6 x 5.6 x 50.8 mm (0.22 x 2.0 in.), and notch depth of 2.3 - 3.5 mm and width of 0.15 mm.

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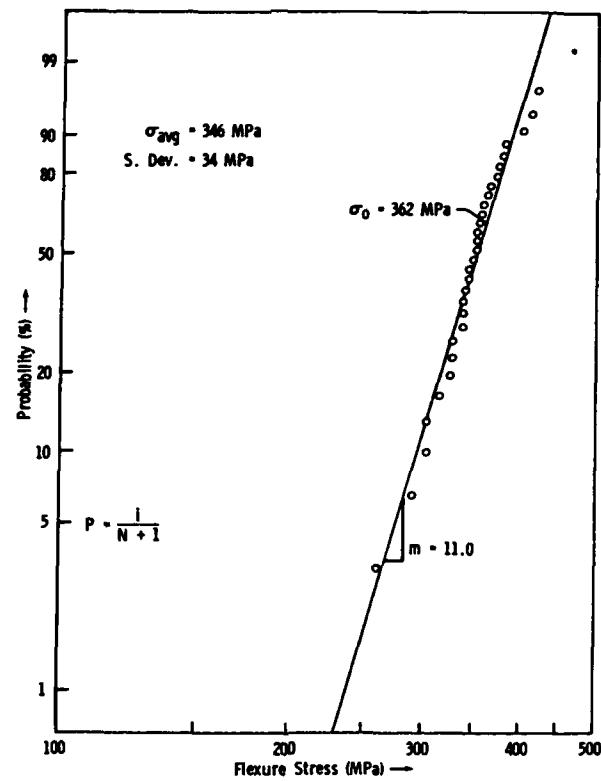


Figure 4. Weibull plot for the reference flexural strength for this study.

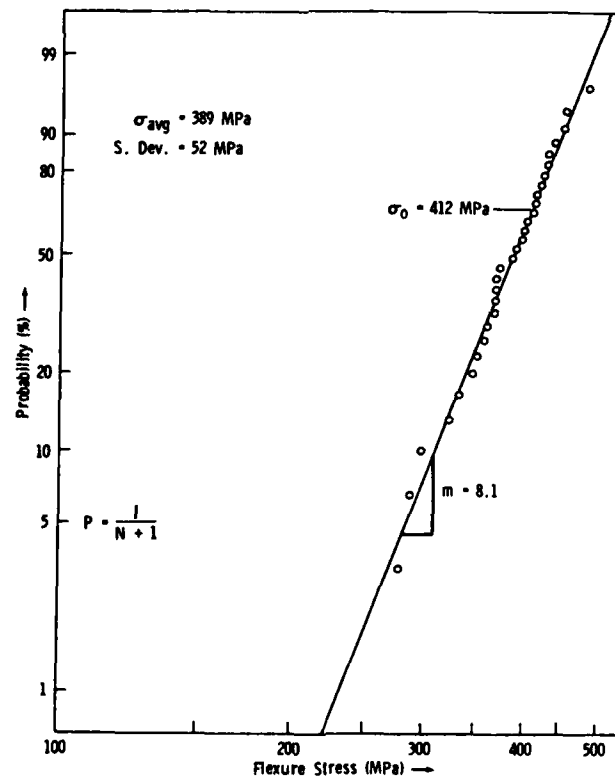


Figure 5. Weibull plot for the parallel study underway at MTL.

All specimens were subjected to a fractographic examination at magnifications of about 100X. Typical fracture surfaces were then photographed and prepared for scanning electron microscopy (SEM) examination. The strength-limiting flaws common to the room temperature flexure specimens were: agglomerates and porous zones either at the surface or distributed within the volume (Figure 6); a few large grains and pores (Figure 7); and an occasional processing crack (Figure 8). Thus, although the Weibull plot implies a single flaw population, there are, in fact, several.

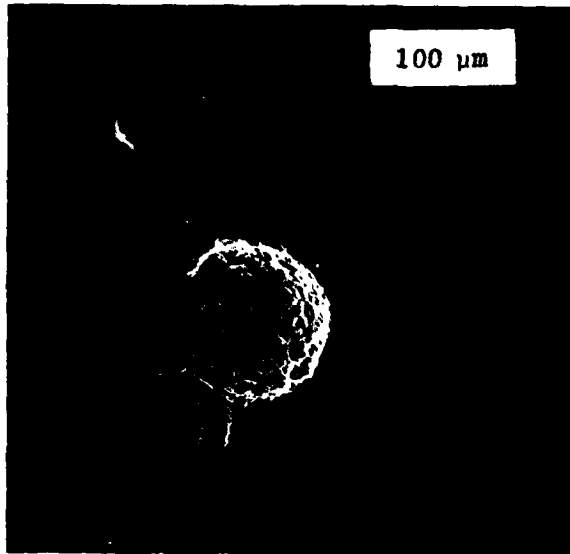


Figure 6. Fracture surface of a control sample that failed due to the agglomerate and pore: 352 MPa.

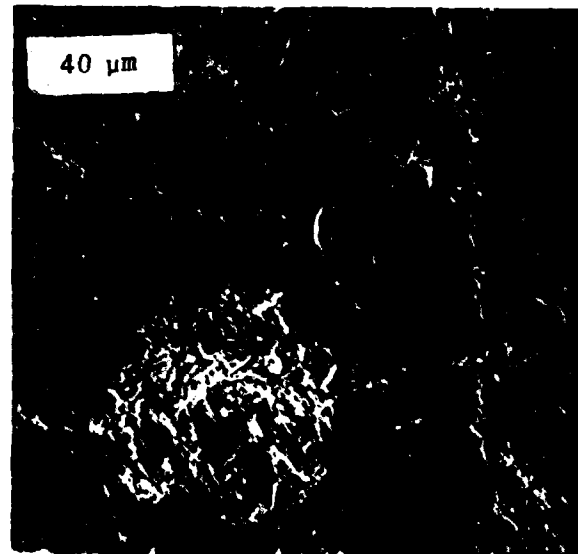


Figure 7. Fracture surface of a control sample: 356 MPa. The strength-limiting flaw is the combination of the porous area and the grain.

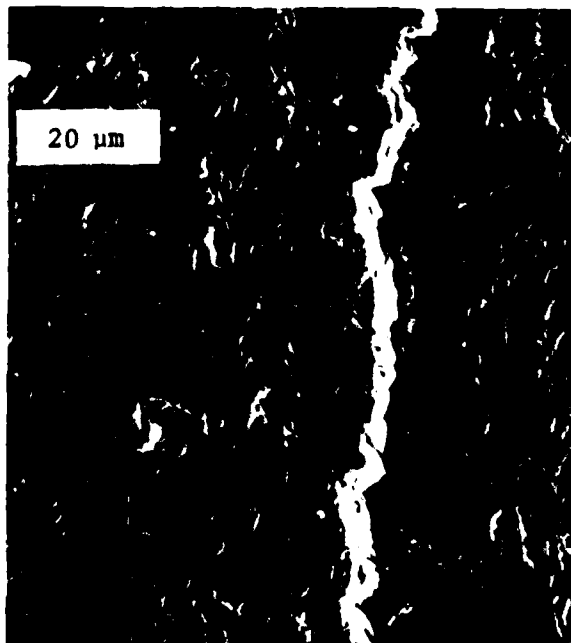


Figure 8. Tensile surface of a control sample: 341 MPa. The crack runs along the longitudinal axis of the specimen and had an overall length in excess of 4.5 mm.

Twelve specimens were tested via the stepped temperature stress-rupture (STSR) procedure and the results are shown in Figure 9. Two specimens failed on loading with applied stresses of 350 and 400 MPa. Four specimens survived the complete cycle with applied stresses of 250 (2), 300 (1), and 325 MPa (1). One specimen survived intact at 350 MPa when an inadvertent microswitch trip shut the furnace down after 22.9 hours at 1400°C. The five remaining specimens with applied stresses of 325 (1) and 350 MPa (4) failed in a time-dependent manner at 1000, 1100, 1200, and 1400°C. Permanent deformation of the five survivors was negligible ($< 0.1\%$ strain). The retained strengths of the four survivors were surprisingly consistent at 401, 448, 402, and 400 MPa for the 250, 250, 300, and 350 MPa STSR applied stresses, respectively. The retained strengths are appreciably higher than the reference room temperature strengths (346 MPa average). This may either be due to a flaw healing process or crack blunting.

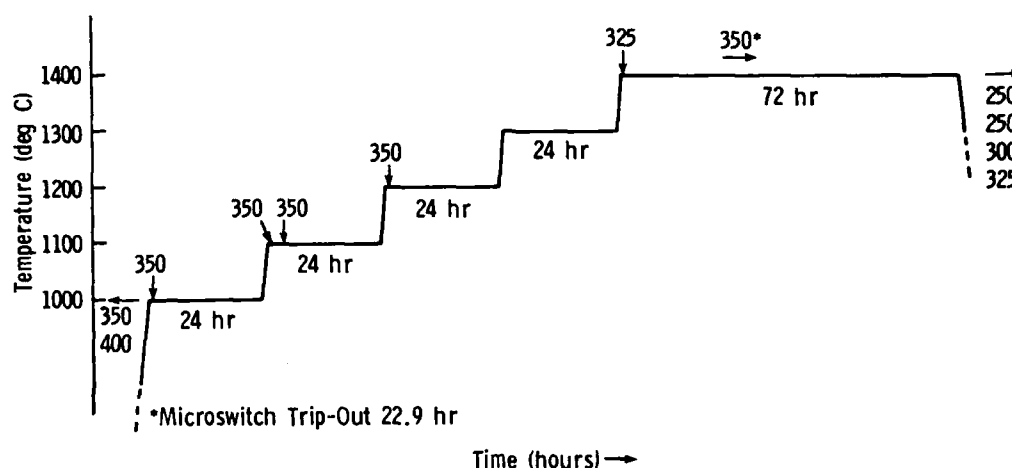


Figure 9. Stepped temperature stress-rupture results.

Twenty-four specimens were tested at 1200°C in the stress-rupture experiments (Figure 10). Nine failed on loading at applied stresses of 350 (2), 400 (6), and 450 MPa (1). Five specimens survived 1000 hours at stresses of 200 (1), 250 (2), and 300 MPa (2). One specimen survived 300 MPa for 52.5 hours when the furnace malfunctioned and shutdown. All surviving specimens had negligible permanent deformation ($< 0.1\%$ strain) indicating an excellent creep resistance. The retained strengths for the 1000-hour survivors were 362, 403, 433, 524 and 524 MPa for the 200, 250, 250, 300, and 300 MPa stress rupture applied stresses, respectively. One specimen which was loaded to 200 MPa survived 10,000 hours without failure. Its retained strength at room temperature was 417 MPa and it fractured from a sintering defect. There was only a slight weight change in the specimen, since the oxide layer which formed was only 0.005 mm thick. The retained strength of all the 1200°C survivors was greater than the average room temperature strength. The remaining eight specimens failed in a time-dependent manner from 0.001 hour to 210.5 hours at applied stresses of 300 and 350 MPa. A static fatigue limit may exist between 200 and 250 MPa. The boundary lines on the stress-rupture plot (Figure 10) were drawn to approximate the scatter. None of the specimen fracture surfaces showed any signs of slow crack growth.

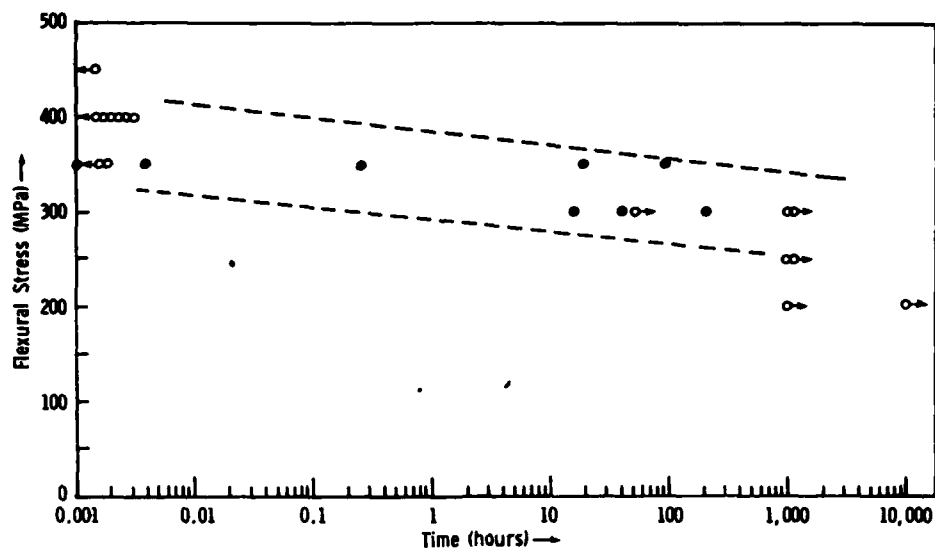


Figure 10. Flexural stress-rupture at 1200°C. The boundary lines approximate the scatter.

The fracture surface characteristics of the stress-rupture and STSR time-dependent failures appear identical to those of the fast fracture specimens. The strength-limiting flaws for the failure on loading specimens are volume distributed and are agglomerates (Figure 11), porous zones, or large grains. The time-dependent failures occurred exclusively from surface-connected porosity (Figures 12 and 13). This porosity was often an area of small interconnected porosity and not necessarily a discrete void.

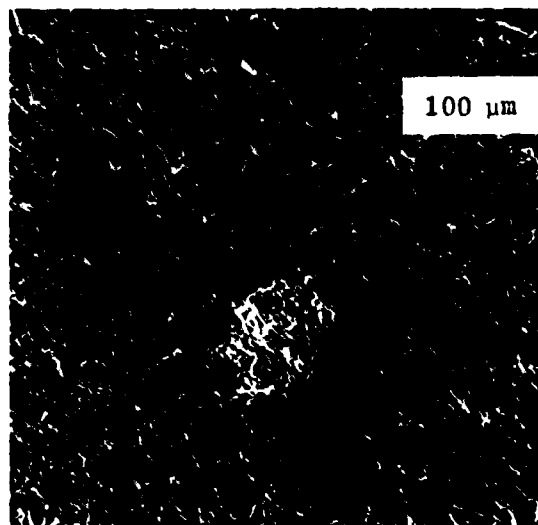


Figure 11. Fracture surface of a failure on loading during a stress-rupture test: 350 MPa. The flaw is a porous area away from the tensile surface.

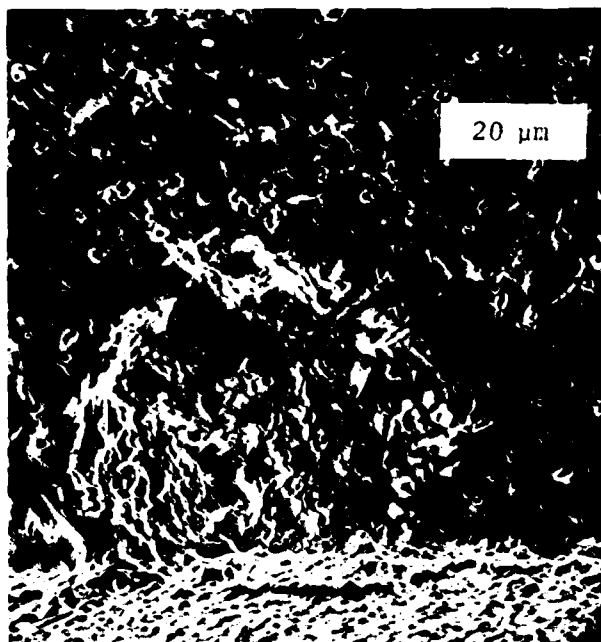


Figure 12. Fracture surface of a time-dependent failure during a stress-rupture test: t : 210 hours, 300 MPa. Failure occurred at a surface-connected porous area.

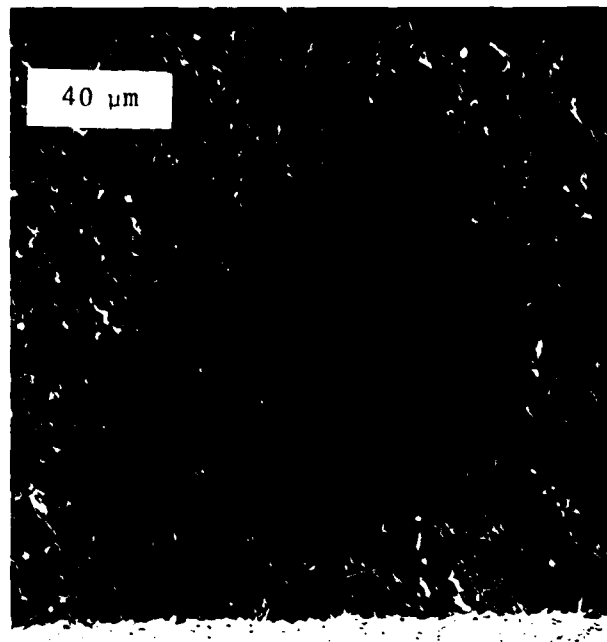


Figure 13. Fracture surface of a time-dependent failure during a STSR test: 325 MPa, 0.4 hour at 1400°C. Failure occurred due to the porous area at the surface which is coincident with a large grain.

DISCUSSION

The beta silicon carbide material is subject to static fatigue failure over the 1000°C to 1400°C temperature range for applied flexural stresses of 300 and 350 MPa, which is at least 85 percent of the reference fast fracture stress. The scatter in the time-to-failure of identically loaded specimens was up to five orders of magnitude. The good strength retention at high temperature and the static resistance findings of this study are consistent with earlier reports.⁷⁻¹⁰ Double torsion experiments on this material failed to detect any evidence of a time-dependent failure process.¹⁶ That time-dependent failure can occur from surface-connected porous zones as shown in this report, underscores the desirability of using stress-rupture testing.

Although this form of silicon carbide is nominally the beta form, the strength-limiting flaws, the favorable creep and static fatigue resistances, and the enhanced strength of survivors are very similar to the results on the sintered alpha form of commercial silicon carbide.¹⁸⁻²⁰ Indeed, the two materials share a susceptibility of time-dependent failure to primarily surface-connected defects, which suggests a stress corrosion phenomena. Controlled atmosphere experiments would be valuable in clarifying this but were beyond the scope of this screening study.

*Carborundum Corporation, Niagara Falls, NY

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20. QUINN, G. D., and KATZ, R. N. *Time-Dependent High-Temperature Strength of Sintered α -SiC*. J. Am. Cer. Soc., v. 63, no. 1-2, January-February 1980, p. 117-119.

CONCLUSION

The General Electric sintered beta silicon carbide tested for this program is of a limited production scale and may not necessarily be identical with earlier research laboratory produced vintages. The Vickers and Knoop microhardness and the Vickers fracture toughness are lower than previously published data.⁷ The room temperature flexural strength is somewhat lower than reported.⁹⁻¹⁰

The intrinsic flaw populations are generally agglomerates and porous zones with a few large grains, inclusions, and process cracks. These flaws are all volume distributed. Time-dependent failures occurred only from surface-connected porous zones. There may be an effective stress-rupture limit at 1000 hours of approximately 250 MPa ($\approx 70\%$ of the fast fracture reference strength) at 1200°C. Time-dependent failures occurred throughout the 1000°C to 1400°C temperature range.

The C-scan promises to become a simple, powerful, and effective quality control test to find not only large property variations within a tile or component, but microstructural variations and severe flaw locations. More in-depth work must be done on the latter two topics for different materials and more complex shapes.

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NOMENCLATURE

V_L	Longitudinal Velocity
d	Thickness
t	Time of flight
E	Elastic Modulus
ρ	Density
ν	Poisson's ratio
E_0	Elastic modulus at theoretical density
P	Porosity
b	Porosity correction factor
ρ_{TH}	Theoretical density

APPENDIX

The ultrasonic C-scan maps the point-by-point wave propagation time through a material. The general equation for the longitudinal wave velocity is

$$V_L = \frac{2d}{t} = \left[\frac{E}{\rho} \frac{(1-\nu)}{(1+\nu)(1-2\nu)} \right]^{\frac{1}{2}} \quad (1)$$

which solving for the propagation time becomes

$$t = 2d \rho^{\frac{1}{2}} E^{-\frac{1}{2}} \left[\frac{(1+\nu)(1-2\nu)}{(1-\nu)} \right]^{\frac{1}{2}} \quad (2)$$

where d, ρ, E , and ν are the tile thickness, density, elastic modulus, and Poisson's ratio, respectively.

The C-scan shown in Figure 1 shows (discounting wave distortion at the edges) the wave propagation time, or fly time, increases by approximately 5.5 percent from the edge to the center of the tile. Because of this large variation in fly time, it was decided to cut five rectangular blocks through the center of the tile to find the effect of the local material properties on the fly time.

The tile, as supplied had a thickness increase from the edge to the center of 1.5 percent. The five blocks had their densities measured (Archimedes' method) and were ultrasonically evaluated for the local elastic modulus and Poisson's ratio. The data are shown in Table A-1.

Table A-1. DENSITIES AND SONIC PROPERTIES FOR
THE MACHINED BLOCKS

Block No.	Density (g/cc)	ρ/ρ_{th}	Porosity (P)	Elastic Modulus, E (GPa)	E/E_0	Poisson's Ratio (ν)
1	3.10	0.967	0.033	404.0	0.907	0.17
2	3.07	0.956	0.044	389.6	0.875	0.16
3	3.05	0.951	0.049	384.4	0.863	0.16
4	3.06	0.954	0.046	388.3	0.872	0.16
5	3.11	0.968	0.032	405.9	0.911	0.16

$\rho_{th} = 3.21 \text{ g/cc}$

$E_0 = 445 \text{ GPa}$

A decrease in the density coincident to a decrease in the elastic modulus from the edge to the center of the tile is readily apparent. This relationship, as described by Rice,²¹ between the elastic modulus and porosity, yields a porosity correction factor (b) of 3.1 and 2.8 for the exponential and linear equations,⁶ respectively, assuming an elastic modulus at zero porosity of 445 GPa (64.6×10^6 psi).^{*} There is no apparent trend in Poisson's ratio versus density through the tile.

This brief analysis does not take into account any secondary microstructure effects on the material properties, such as grain size. It was assumed that the material is homogeneous and the porosity is directly related to density ($P = 1 - \rho/\rho_{TH}$).

The C-scan map showed a change for 4.0 percent from block 1 to 3. Using the local properties of the blocks with Equation 2, the change in fly time is +3.6 percent. This shows an outstanding agreement even through the "local" properties are themselves averaged. The computations are detailed below.

ANALYSIS OF THE C-SCAN

The 4% slower times at the center (3) versus the edge (1) from the C-scan suggest: $\frac{t_3}{t_1} \approx 1.040$.

Table A-2. EFFECT OF INDIVIDUAL VARIABLE UPON RATIO t_3/t_1 .

	Block #1 (Edge)	Block #3 (Center)	$\Delta\%$ #1 \rightarrow #3	$\frac{t_3}{t_1} = \dots$
d (cm)	1.3510	1.3706	+1.45%	1.0145
ρ (g/cm ³)	3.10	3.05	-1.59%	0.9920
E (GPa)	404.2	384.7	-4.85%	1.0252
ν	0.17	0.16	-2.94%	1.0046

$$\frac{t_3}{t_1} = \frac{d_3}{d_1} \left(\frac{\rho_3}{\rho_1} \right)^{1/2} \left(\frac{E_1}{E_3} \right)^{1/2} \left(\frac{F(\nu_3)}{F(\nu_1)} \right)^{1/2} \quad \text{with } F(\nu_i) = \frac{(1+\nu_i)(1-2\nu_i)}{(1-\nu_i)}$$

$$= (1.0145) (0.9920) (1.0252) (1.0046) = 1.036$$

$$\underline{1.040 \approx 1.036}$$

C-Scan Local Properties

$$\frac{E}{E_0} = e^{-3.1P} \quad \text{and} \quad \frac{E}{E_0} = 1 - 2.8P$$

21. RICE, R. W. *Microstructural Dependence of Mechanical Behavior of Ceramics* in *Treatise of Materials Science and Technology*, R. K. MacCrone, ed., v. II, Academic Press Inc., NY, 1977, p. 203-230.

A breakdown on the change in properties required to cause an increase in the fly time is:

Increased thickness	- Major contribution
Decreased elastic modulus	- Minor-major contribution
Decreased Poisson's ratio	- Negligible contribution

Note that a decrease in density, coincident with a decrease in elastic modulus, will decrease the fly time.

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